

mathematical model of relaxation liquid phase epitaxy with mass-transport reversal.

5. In summary, the proposed and experimentally verified model of relaxation liquid phase epitaxy with mass-transport reversal enables us to account for the substantial differences in the mechanisms of the initial stages of layer growth on the main and reversing substrates in the process of relaxation of the supersaturation of the melt, which makes it possible to control the thicknesses of sub-micron layers.

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Translated by S. A. Stewart

Pulsed vacuum-UV irradiation of AsSe and As₂S₃ glassy chalcogenide semiconductor films

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(Submitted April 25, 1991)

Zh. Tekh. Fiz. **62**, 107-114 (March 1992)

The effect of pulsed radiation from an ArF excimer laser ($\lambda = 193$ nm, $\tau = 20$ ns) on glassy chalcogenide semiconductor films of As₂S₃ and AsSe is investigated. Photodarkening, photoablation, and photothermal ablation are observed in succession as the radiation flux density is increased to 100 mJ/cm². The intensity of the photodarkening increases sharply as λ_0 moves to the vacuum UV region and has a nonlinear character: the reciprocity law fails. Moreover, the photodarkening is accompanied by a change in the chemical solubility of the irradiated parts of the material. Photoablation begins at 20-25 mJ/cm². The dependence of ablation speed on pulse energy E_p has two parts: in the first part photoetching occurs immediately, and in the second ($E_p > 50$ mJ/cm²) photothermal ablation prevails. Use of these effects to record images can lead to high values of contrast $\gamma \approx 10$, which, in the case of vacuum-UV photolithography, provides the possibility of attaining submicron resolution.

1. Photostimulated transformations in glassy chalcogenide semiconductor (GCS) films are traditionally studied using visible radiation,¹ in particular, as applied to the use of GCS as photoresists in microelectronic technology (for example, Ref. 2). Such films are practically never studied using pulsed laser radiation in the vacuum UV region. At the same time, a reduction in the wavelength λ_0 of the radiation incident on the film into the vacuum UV region of the spectrum might lead us to expect qualitative changes when the condition $h\nu > E^*$ is satisfied, where E^* is a certain critical energy, e.g., the energy of chemical binding or atomization.

The GCS films we studied were of As₂S₃ and As_{0.5}Se (the latter is denoted below by AsSe), obtained by thermal evaporation and deposition in vacuum on silicon and glass substrates. The films were $h_0 = 0.1-0.3$ μ m thick. A pulsed excimer laser ELI-72 working at wavelengths 308, 248, and 193 nm, pulse length 20-30 ns, and repetition rate 10-150 Hz was used as a radiation source. The dimensions of the output beam and the maximum energy per pulse were $S = 10 \times 20$ mm² and 150 mJ respectively. Uniformity of illumination in

the plane of exposure was assured by use of a quartz condenser based on diffraction grating lenses. The experiments were mainly carried out near the long-wavelength boundary of the vacuum UV region, at $\lambda_0 = 193$ nm, in ArF excimer molecule radiation.

2. Figure 1 shows transmission and reflection spectra of an AsSe film before and after irradiation by a single pulse at $\lambda_0 = 193$ nm and $E_p = 50$ mJ/cm². The spectra were taken on samples deposited on substrates of monocrystalline MgF₂ grown in vacuum especially for use in the vacuum UV region. A two-beam vacuum spectrophotometer with a low-power gas-discharger hydrogen source was used. The spectra before irradiation are completely typical for thin GCS films: 0.3 μ m films are practically opaque for $\lambda < 500$ nm, since $\alpha \geq 10^5$ cm⁻¹ down to $\lambda \sim 200$ nm. As regards the region of transparency ($h\nu < E_g$), the first orders of interference are observed here, depending on the thickness h_0 . The spectra after irradiation clearly display the photostimulated changes in the optical properties of the film. In the region of transparency the well-known¹ relative photodarkening is observed, and this is

apparently related to some reduction in the interference amplitude in the transmission spectra and, especially, in the reflection spectra. A weak shift in the interference maxima can also be seen. However, changes observed in the fundamental band in the region of relative bleaching, at $\lambda \sim 200$ nm, near the laser wavelength, are the most interesting feature, and require further study.

The dependence of the relative darkening $\delta T = (T(H) - T(0))/T(0)$ ($T(0)$ and $T(H)$ are the transparency before and after irradiation, respectively) on the vacuum UV radiation dose H , measured at $\lambda_c = 630$ nm, is shown in Fig. 2. Photodarkening is noticeable even at a few mJ/cm². Such high sensitivity (more than three orders of magnitude higher than for exposure to continuous laser radiation at wavelength $\lambda_0 = 514$ nm; for comparison, see Ref. 3), might be attributed to local heating of the film by the powerful radiation pulse, including a thermal mechanism of photostimulated reactions, which turns out to be more efficient compared to the phototransport reactions that occur even under low-intensity continuous irradiation. However, studies show that the spectral composition of the radiation has an important influence on the efficiency of the reaction, an indication of the nonthermal nature of the phenomenon. For example, for pulses of laser radiation of identical peak power and duration, we had to use 10 times the exposure at 308 nm as for the vacuum UV radiation from the ArF laser

(193 nm) to produce the same degree of photostimulated darkening of a film of AsSe, as measured at wavelength $\lambda_c = 630$ nm.

The dependence of T on H is linear up to $H \sim 20$ -30 mJ/cm², and thereafter completely saturates at $T \sim 0.2$, and upon reaching $H \sim 1$ J/cm² falls sharply as the mechanism changes to photobleaching. Experiments were made with pulses of fixed energy E_p and the accumulated dose resulting from N pulses was taken to be $E_p \times N$. When the pulse energy is increased from 2 mJ/cm² (curve 1) to 4 mJ/cm² (curve 2), the photodarkening becomes more intense and reaches saturation at somewhat lower exposures. Thus with nanosecond pulses of vacuum UV radiation the reciprocity law fails, that is, the photodarkening depends not only on dose but on pulse energy.

Similar "nonlinearity" of the photosensitivity of GCS films was observed in the visible region by Mednikarov,² where, moreover, its positive effect of increasing the resolving power was noted.

The tendency towards "bleaching" of GCS films at large doses (number of pulses $N > 500$) is evidently due to the fact that the corresponding parts of the curves were obtained in the multipulse regime of exposure. In this regime, first, the period of pulse repetition $t_r = 1/\nu$ is comparable with the dark relaxation time of photoexcitation (for As-Se compounds it is⁴ about 10^{-1} - 10^{-2} s), and there is insufficient time for a quasistationary regime of photodarkening to be established. Second, an increase in dose H leads to photoablation, the immediate removal of part of the material by the radiation. As N and ν increase, these processes accelerate and the result is local heating, so that the thickness decreases during exposure. Thus, for complete ablative vacuum-UV etching of a 0.2 μ m AsSe film for $E_p = 4$ mJ/cm² and $\nu = 50$ Hz, we needed to use 1250 pulses or an exposure time $\tau = 25$ s, which corresponds to 0.16 nm per pulse.

3. The high sensitivity to vacuum UV radiation, evidently accompanied by certain structural changes, suggests the use of these films as a sensitive resist for vacuum-UV laser lithography; the production of optical images under reduced illumination contrast is an attractive possibility, due to the "nonlinearity" of the optical properties. To study GCS as a vacuum-UV resist, after exposure the films were developed in monoethanolamine, using it as a negative developer. Figure 3 is a plot of the exposure threshold H_0 corresponding to complete formation of a negative image, as a function of the pulse ener-

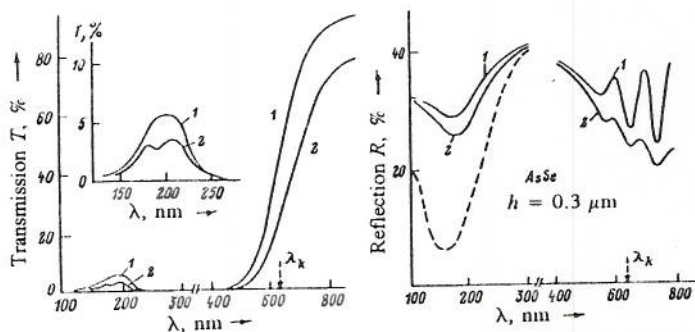


FIG. 1. Averaged spectra in (a) transmission and (b) reflection for a 0.3 μ m AsSe film: 1) before irradiation; 2) after irradiation, $E_p = 50$ mJ/cm², $\lambda_0 = 193$ nm. The dashed arrow on the abscissa is at λ_c , where the control measurement was made. The dashed curve is the change in reflection after prolonged irradiation by a complex spectrum of continuous vacuum-UV radiation from a source of the GIS type. The interference maxima are excluded from the transmission spectra. Measurements were made at flux densities not exceeding 10^{-6} J/cm².

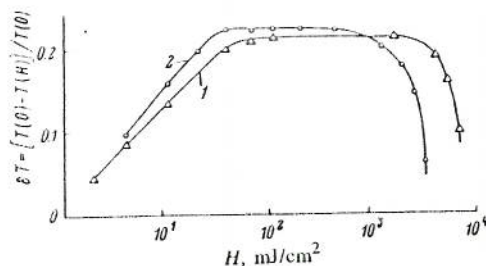


FIG. 2. Photodarkening δT in AsSe films as a function of the exposure H : ($h = 0.2$ μ m), 10 Hz, 20 ns, $\lambda_0 = 193$ nm; 1) $E_p = 2$ mJ/cm², 2) $E_p = 4$ mJ/cm².

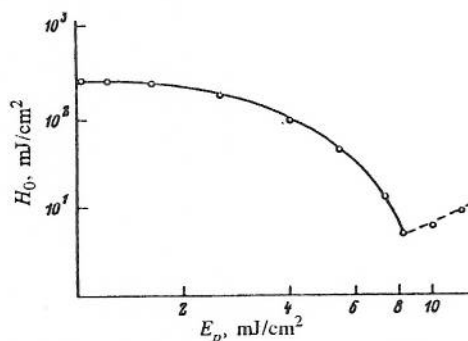


FIG. 3. Exposure threshold H_0 as a function of laser pulse energy E_p ($\lambda_0 = 193$ nm) for AsSe films 0.2 μ m thick; developed in monoethanolamine for 5 min.

gy of an ArF excimer laser. The function is shown for a 0.2 μm film of AsSe after 5 minutes development. For small doses (peak power up to 0.2-0.25 mW/cm^2) the threshold exposure changes slightly and is about 350 mJ/cm^2 . Beginning at $E_p = 1 \text{ mJ}/\text{cm}^2$ a gradual reduction in H_0 is observed, reaching 50% for $E_p = 5 \text{ mJ}/\text{cm}^2$, and only one pulse is required for full development of the drawing at energy $E_p = 8 \text{ mJ}/\text{cm}^2$. Further increase in the pulse energy does not change the situation, and the increase in H_0 in Fig. 3 only means that more energy is expended in the pulse.

In our experiments with liquid developing we could not reproduce elements of the drawing with dimensions less than 2.0 μm for thicknesses h_0 greater than 0.25 μm , that is, the ratio $h_0/\Delta a$, where Δa is the blurring of the figure, did not exceed 0.1; this is poorer performance than for industrial technology using organic resists. The lowering of the limiting resolution of GCS achievable in a two-stage variant of the photolithographic process with liquid developer is evidently a result of degradation of the latent image due to the surface character of the absorption. In fact, measurements of the absorption coefficient α of thin layers in the vacuum UV range give values of $(1.5) \cdot 10^5 \text{ cm}^{-1}$, which limits the penetration of vacuum UV photons to a depth of only several nanometers. This is important primarily for the negative process, since an insufficient change in the solubility of the lower layers results in undercutting of the image formed in the upper layer destruction of the negative drawing. This shortcoming appears to be less important when processing films in "positive" developer, and to a lesser degree for As-S compounds than for As-Se.

4. As the pulse energy E_p increases, along with photodarkening of the GCS films, photoablation becomes more intense, and beginning at $E_p = 20\text{-}25 \text{ mJ}/\text{cm}^2$, etching of an AsSe layer as a result of the radiation becomes visually apparent after exposure to a single pulse. The experimentally determined photoetching speeds $U_{pe}(E_p)$ for AsSe and As_2S_3 are plotted in Fig. 4 on a semilogarithmic scale.

The function $U_{pe}(E_p)$ for GCS films for the spectral ranges studied can be approximated by two exponential parts with a transition region between them in the region $E_p = 50\text{-}60 \text{ mJ}/\text{cm}^2$.

The appearance of a sharp bend in the characteristics may be due to an additional mechanism of immediate photoablation

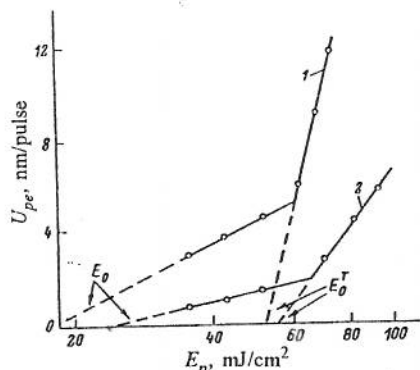


FIG. 4. Circles are the measured speeds of vacuum-UV etching of 1) As_2S_3 and 2) AsSe films as a function of pulse energy E_p of an ArF excimer laser (193 nm). Curves are: 1) $1.8\ln(E_p/22) + 11\ln(E_p/55)$; 2) $3\ln(E_p/13) + 41\ln(E_p/52)$; for $E_p < 50\text{-}60$ the first terms predominate, and for higher energies the second terms predominate; E_p is in mJ/cm^2 and U_{pe} in nm/pulse . The arrows show the values of the critical flux densities.

(photodecomposition or photooxidation: the experiments were conducted in air) with a thermal component. Its influence may become important, since, as a result of the short radiation pulses, the energy distribution of the phonons is far from the equilibrium distribution, that is, the local increase in the effective temperature may significantly exceed the value calculated for equilibrium thermodynamic characteristics.⁵ In such a case for the description of the changes in thickness we can use the expression (henceforth we consider it necessary to supplement the formulas from Ref. 6, which were derived for polymer films, with a coefficient $(1 - R)$, which takes into consideration the reflection of radiation from the surface of the GCS film):

$$h = h_{ph} + h_T = (1/\alpha) \ln[(E_p/E_0) \times (1 - R)] + A \exp(-W/kT). \quad (1)$$

The first term describes nonthermal photoablation with threshold energy E_0 , and the second describes thermal photoablation with a local temperature T proportional to E_p and with an activation energy W of the chemical reaction. The low-energy slopes of the curves in Fig. 4 give $\alpha = 9 \cdot 10^5 \text{ cm}^{-1}$ and $4 \cdot 10^5 \text{ cm}^{-1}$ for AsSe and As_2S_3 , respectively. This exceeds the values measured in transmission on a spectrophotometer, but agrees with the observed fact of photodarkening under vacuum-UV radiation. Considering the volume heated by the laser pulses to be limited to the depth of photon absorption $l \sim \alpha^{-1}$, and neglecting the temperature of the medium, we get for the thermal component h_T :

$$h_m = A \exp \{ -(B/\alpha) \ln[(E_p/E_0^T) (1 - R)] E_p^{-1} \}, \quad (2)$$

where E_0^T is the threshold energy at the start of the thermal process, and $B = W/kC\rho$, where k is Boltzmann's constant, C is the specific heat, and ρ is the density of the material.

Plots of h_T are shown in Fig. 5. The experimental data are satisfactorily "linearized" in corresponding coordinates, permitting an estimate of the constants $A = 0.05$ and $0.2 \mu\text{m}$ (corresponding to the thicknesses at which the speed of the thermochemical reaction saturates) and $B/\alpha = 0.17$ and $0.12 \text{ J}/\text{cm}^2$ for AsSe and As_2S_3 , respectively. On the other hand, we can immediately make a tentative determination of the temperature at the start of the process, using the estimate:

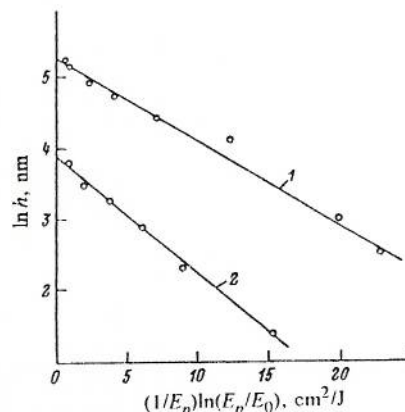


FIG. 5. Speed of photoetching in the thermal region (thickness h_T , etched by one pulse) as a function of pulse energy density in coordinates corresponding to equation (2) for films of: 1) As_2S_3 2) AsSe: $\lambda_0 = 193 \text{ nm}$.

TABLE I. Technological Parameters of GCS Films with Vacuum-UV Etching, $h_0 = 0.2 \mu\text{m}$

E_p , mJ/cm ²	AsSe				As ₂ S ₃			
	H_0 , J/cm ²	$\partial h/\partial H$, 10 ⁻⁷	$H_0 \partial h/\partial H$, μm	γ_{eff}	H_0 , J/cm ²	$\partial h/\partial H$, 10 ⁻⁷	$H_0 \partial h/\partial H$, μm	eff
25	7.7	0.28	0.23	2.8	2.4	0.8	0.20	2.1
50	7.5	0.35	0.26	3.1	2.2	1.6	0.36	4.3
60	6.6	0.70	0.46	5.4	1.8	2.5	0.45	8.6
70	5.6	1.0	0.56	5.7	1.3	4.0	0.50	9.4
80	2.8	2.2	0.62	6.2	0.8	7.0	0.56	10.5

$$T = \alpha E_0^T (1 - R) / C \rho, \quad (3a)$$

or for $\delta > \alpha^{-1}$,

$$T = E_0^T (1 - R) / C \rho \delta, \quad (3b)$$

where $\delta = (K\tau/\rho C)^{1/2}$ is the thermal diffusion length and K is the thermal conductivity. For GCS the thermal diffusion length is of the order $\delta \sim 0.1 \mu\text{m}$, which is equal to greater than $l = \alpha^{-1}$, and the instantaneous temperature at the start of the photothermal ablation turns out to be of the order of $4 \cdot 10^3 \text{ K}$.

It is interesting that for the range of E_p studied, arsenic sulfide etched photoablatively 3.5-4.5 times more intensely than the selenide.

5. Introducing the parameter γ_{eff} , analogous to the developing factor in the two-stage photolithographic process, we can calculate its value immediately from the function $U_{pe}(E_p)$ using the relation:

$$\gamma_{\text{eff}}(E_n)_p = U_{pe}^{-1}(E_n) \cdot dU_{pe}/(dE_n). \quad (4)$$

The results of the calculation are shown in Table I. The values of γ_{eff} are very large, and are difficult to realize in traditional photolithography, either with organic positive photoresists⁷ or with inorganic ones,² and are comparable with those obtained through use of contrast-enhancement layers in multilayer photoresist systems.⁸ This shows the large limiting resolution potentially possible with GCS.

By virtue of the relatively low photosensitivity of GCS films used a self-developing vacuum-UV resists in the ablation regime (Fig. 6 and Table I), the transfer function $\partial h/\partial H$ is ~ 2 orders of magnitude smaller than for vacuum-UV etching of organic films. At the same time the transfer function obtained is 5-10 times larger than that obtained for the other inorganic vacuum-UV resist, the "diamond-like" film, which holds the known record resolution of 0.13 nm for excimer laser lithography.⁹

Figure 6 shows the measured exposure threshold H_0 necessary for full photoablation vacuum-UV etching of GCS films in the single-pulse regime ($t \gg 0.1 \text{ s}$), as a function of the ArF excimer laser pulse energy. Comparing curve 1 in Fig. 6 with Fig. 3 we see that the single-stage process of formation of the relief image (the positive) occurs for values of H_0 substantially larger than for the two-stage (negative) process with the liquid developer, and that its sensitivity increases, although to a lesser degree, with an increase in E_p . Vacuum-UV etching requires a 50-75 times larger exposure H_0 , although as a practical matter the exposure times remain very small and do not exceed several seconds for $\nu = 30\text{-}50 \text{ Hz}$. As to the more intensive regime for As₂S₃, which is realized for $E_p = 80 \text{ mJ/cm}^2$, complete removal of a 300 nm layer

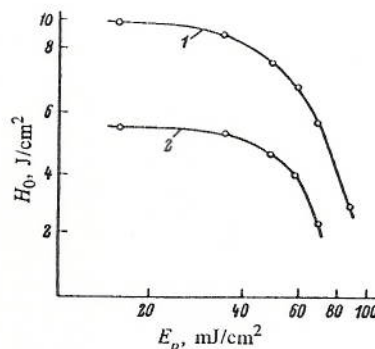


FIG. 6. Exposure threshold H_0 as a function of laser pulse energy E_p ($\lambda_0 = 193 \text{ nm}$) for vacuum UV etching of GCS films: 1) $0.2 \mu\text{m}$ AsSe, 2) $0.3 \mu\text{m}$ As₂S₃. Single-pulse regime, interval between pulses $t \gg 0.1 \text{ s}$.

required ~ 10 single pulses, that is, the speed of etching was 30 nm/pulse . This means that the single-pulse regime is quite realistic for multilayer photoresist systems, where the upper layer of GCS forming the picture is made no thicker than $30\text{-}40 \text{ nm}$.

It is easy to see that for the pulsed regime of exposure, in the general case of nonthermal ablation (the multipulse case), one has

$$H_0(\partial h/\partial H) = h_0. \quad (5)$$

Table I lists values of the product $(\partial h/\partial H)H_0$, which for flux densities $E_p < 50 \text{ mJ/cm}^2$, that is, in the "nonthermal" region, agree within experimental error with the measurements, that is, they are equal to the thickness of the film, but thereafter increase rapidly.

It is worth keeping in mind that, in the practical use of GCS films as self-developing vacuum UV resists, completely "dry" technological processes in exclusively oxygen-free media are preferred, which, besides having other virtues,¹⁰ avoids the creation of highly toxic oxides.

We performed direct photolithographic experiments on GCS to obtain microdiagrams of integrated circuits. The best resolution was $0.6 \mu\text{m}$ and was limited by the phototemplate used. The quality of the microdiagram obtained shows that this is not a limiting value, and higher resolution is possible.

6. This study has established a number of features of the behavior of thin GCS films of AsSe and As₂S₃ under vacuum UV pulsed irradiation.

The most important advantage is the very low dose neces-

sary for noticeable photostimulated changes of the optical properties (of the order of 1 mJ/cm^2) and the high value of the slope $\partial\alpha/\partial H$ or photochromic sensitivity attained in the vacuum UV region of the spectrum, up to 10^{-6} cm/J for $\lambda_0 = 193 \text{ nm}$. The changes in the optical properties are accompanied by photostimulated changes in the etchability, allowing the use of these GCS compounds as highly sensitive inorganic photoresists (8 mJ/cm^2 per $0.2 \text{ }\mu\text{m}$ of thickness in the single-pulse regime with $\tau = 20 \text{ ns}$; this is a record value both for inorganic and organic resists). However, the attainment of high resolution here requires further work on the development process. With increased pulse energy a one-step process (photo-etching) requiring somewhat larger exposures ($\sim 0.5 \text{ J/cm}^2$) is possible. However, this regime is realized for not more than 10 pulses ($\tau_0 < 10 \text{ s}$) at $0.2 \text{ }\mu\text{m}$, but in the case of three-layer resists with a thin GCS upper layer ($h_0 \sim 30 \text{ nm}$) a single-pulse process ($\tau = 20 \text{ ns}$) is again possible, so the exposure can be done "on the fly," without stopping the table. A feature of the photolithographic processes studied is the very high image contrast, due to the photodarkening effect, which is a prerequisite for attaining submicron resolution, better than $0.6 \text{ }\mu\text{m}$.

All the possibilities of application enumerated, obviously,

are based on the structural changes in GCS arising from vacuum-UV irradiation, primarily in the region of relative bleaching at $\lambda \sim 190 \text{ nm}$; their nature requires further study.

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Translated by B. R. Bullard

pulsed excimer laser radiation. At first we investigated the reproduction of stripes with size value equal or less of one Fresnel zone. These values are theoretical inaccessible from the wave optics point of view.

2. EXPERIMENT TECHNIQUE.

In our experiments we used the laboratory equipment which consists of ArF (193 nm) excimer laser with 20 ns pulse duration, quartz condenser using raster diffraction lens, and turning mirror. Thus, the homogeneous illumination ($\pm 20\%$) has been created on a sample area $D=50\text{mm}$. Laser was optically combined with mechanical part of aligner. Two masks have been used for definition of resolution capability. The first was produced by electron-lithography method on quartz substrate coated with chromium. The minimal element size was 300 nm. The second was produced by interference-holographic method on VUV-quality monocrystal MgF_2 substrate coated with gold⁵. The minimal element size was 60-80 nm on different areas. The scanning electron micrograph (SEMs) of one of this areas is shown on Fig.1. The As_2S_3 layers had 800-1000 Å thickness and were deposited in vacuum on polished semiconductor Si or GaAs substrates.

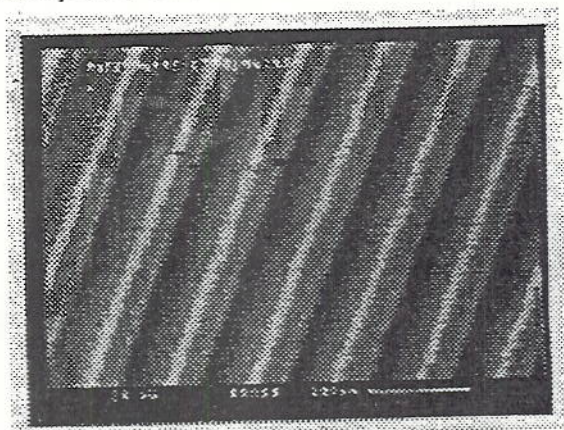


Fig1. Scanning electron micrograph (SEMs) of VUV-mask obtained by interferential method on MgF_2 coated by gold. The minimum dimension is $a_{\min}=80\text{ nm}$.

3. EXPERIMENT RESULTS.

The preliminary experiments were carried out on quartz masks using KrF radiation (249 nm). Fig.2 shows the pattern of high frequency field-effect transistor gate. The width of it is 200-250 nm and the thickness of As_2S_3 film is 200 nm. The pattern was obtained by two 10 mJ/cm^2 pulses in combination with standard wet development. One can see that the details of photomask are good reproduced and edge smearing is about 90 nm. The 300-500 nm elements were reproduced using one pulse of 8 mJ/cm^2 and following wet developing. The smearing in this case does not exceed 70 nm. Apparently some decrease of stripe width (up to 25%) results from diffraction exposure of shadow (Fig.3).